

# **EXHIBIT 90**

## Chapter 5

**AIRBORNE DUSTS**

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brous dusts, the usefulness of such devices for asbestos was explored. Generally it was found that nonrespirable fibers (i.e., on ARC definition, greater than 3  $\mu\text{m}$  in diameter) were sparse and did not contribute much to fiber counts. Also the elutriator fitted to the LRTP was thought to trap some long respirable fibers and so it was removed by the ARC. With the membrane filter method, no elutriator was employed; its addition would have necessitated the use of a fixed flow rate and deprived the method of its flexibility.

There was also exploration of respirable gravimetric sampling, as had taken over for nonfibrous mineral dusts. The mass of total respirable material was, however, judged not to be a good index of the dangerous fiber content, nor could bulk analysis distinguish fibers.

Although selective sampling has not found use for fibers, there would appear to be circumstances where it might be advantageous, for example, for MMMF that contain a high proportion of nonrespirable ( $d > 3 \mu\text{m}$ ) fibers and in "user" situations where nonfibrous coarse dust is also present. It could be expected to provide better discrimination of the respirable material (including fibers) than is given by the  $< 3 \mu\text{m}$  diameter criterion.

### 3. Pathogenic Fibers: "Stanton" Fibers

The foregoing has been concerned with the pulmonary deposition of fibers and other dust particles as a function of size and shape. The subsequent biological reaction, especially to fibers, is also a function of their size and shape. Measurements of airborne dust concentrations made for hygienic purposes should take account of this.

Biological studies have shown that short fibers are readily phagocytosed and rapidly cleared from the lungs; the hazard arises from fibers that are too long to be engulfed by macrophages. The outstanding work of Stanton and Pott and their associates<sup>24,25</sup> has linked carcinogenicity with fibers in the dimensional range: length  $> 8 \mu\text{m}$ ; width  $< 1.5 \mu\text{m}$  (or even  $< 0.25 \mu\text{m}$ ). Carcinogenicity appears to increase with increasing fiber length and with diminishing diameter, but there is no sharp boundary between dangerous and safe particles. Other work has indicated that fibrosis (asbestosis) is attributable to fibers longer than circa 10  $\mu\text{m}$ , thus generally confirming the early studies of Gardner et al. summarized by Vorwald.<sup>19</sup>

Fibers in the dimensional range: length  $> 8 \mu\text{m}$ ; width  $< 1.5 \mu\text{m}$  are sometimes referred to as *Stanton fibers* and have been used by some authors as a basis for the expression of airborne fiber concentrations.<sup>26</sup> Complete detection of such fibers requires the use of electron microscopy.

A further essential characteristic of hazardous fibers is that they must be durable in the body over periods of months or years. Amphibole asbestos is more durable than chrysotile. MMMF varieties differ in durability but are mostly much less durable even than chrysotile fibers.<sup>27-29</sup>

## VI. MEASUREMENT OF ASBESTOS DUST IN THE OCCUPATIONAL ENVIRONMENT

Regulatory standards for fibers in the occupational environment are generally expressed in terms of fibers longer than 5  $\mu\text{m}$  sampled by the membrane filter method (introduced after 1965) and counted by phase contrast microscopy.<sup>2,30</sup> These are commonly referred to as *PCOM fibers* or *regulatory fibers*.

### A. THE MEMBRANE FILTER REFERENCE METHOD FOR ASBESTOS

The sampling of dust or fiber concentrations for microscopical evaluation was greatly simplified by the introduction of membrane filters. These are thin films of dried organic gels having a uniform fine pore structure and high filtration efficiency. The dust sample is collected by drawing a known volume of air through the membrane, usually a 25 mm diameter disc, which is afterwards mounted on a glass slide and made transparent by filling the pores with a medium of matching refractive index or by treatment to collapse the pore structure. The

collected particles are then examined and counted microscopically. For asbestos and MMMF, phase-contrast microscopy is used to increase the visibility of the fibers, as the difference in refractive index between the fibers and the filter material is small.

The method was developed for the measurement of asbestos fibers by the member companies of the ARC circa 1965,<sup>31,32</sup> and soon replaced the LRTP in Britain; the criteria for "countable fibers" (length > 5  $\mu\text{m}$ , aspect ratio > 3:1) remained the same. This was the preferred method of the BOHS (1968) Standard,<sup>33</sup> but minor changes were introduced in the early 1970s.<sup>34</sup> The development and evaluation of an essentially similar method by the U.S. Public Health Service was described in 1968.<sup>35</sup> However, it was said to be unsuitable for the determination of compliance with threshold limit values (TLVs), which continued to be based on particle counts by impinger. Change to a TLV based on the numerical concentration of fibers longer than 5  $\mu\text{m}$ , measured by the membrane filter method, was introduced by the ACGIH in 1970. The Quebec asbestos production industry largely followed U.S. practice.

A major attraction of the membrane filter method of sampling was its essential simplicity. It opened the way for *personal sampling* by instruments that could be "worn" by operatives to collect dust from the breathing zone.<sup>31</sup> Previously, sampling was generally carried out at strategic fixed locations (static sampling).

By the mid-1970s the membrane filter method was in widespread use throughout the world for the measurement of work-place asbestos dust concentrations. However, experience of the method, including exchanges of samples for duplicate evaluation, showed that it did not always produce comparable results when used by different laboratories and by different workers<sup>36,37</sup> (as had applied to particle counts made by earlier methods, thermal precipitator, impinger, etc.). The differences in sample assessment levels (as distinct from differences due to sampling procedures: site, duration, flow rate, etc.) arose from various causes: visibility limit of thin fibers, interpretation of complex particles, and personal factors. The differences between even experienced laboratories could be manyfold. In an attempt to minimize such differences, the Asbestos International Association, after extensive international consultation, published in 1979 the "Reference Method for Determination of Airborne Asbestos Concentration at Workplaces by Light Microscopy (Membrane Filter Method)".<sup>2</sup> This describes materials and procedures in great detail and gives diagrams illustrating how numerous varieties of complex particles are to be enumerated. It was the basis of the European Reference Method adopted by the Council of the European Communities in 1983,<sup>30</sup> and given effect by the Health and Safety Executive in Britain.<sup>38</sup> In the United States, the standard USPHS/NIOSH Membrane Filter Method for Evaluating Airborne Asbestos Fibers, issued in 1979,<sup>39</sup> differed only in some small respects from the European method.

The smallest diameter of fiber detected by the membrane filter/phase contrast optical microscope method has been variously estimated as between 0.2 and 0.4  $\mu\text{m}$ ; circa 0.25  $\mu\text{m}$  should be attained when the reference methods and control procedures are properly applied.

Experience has shown that written instructions are insufficient to ensure comparable counting levels by different observers. Uniform performance requires regular exchange of samples and cross-checking between observers, and the use of permanent standard samples to ensure long-term stability. Accreditation and check-counting quality control schemes are now in operation in America and Europe.<sup>39,40</sup> Machine-aided counting is now sometimes employed and should help to preserve long-term stability in the counting levels of laboratories participating in quality control schemes.<sup>41</sup>

## B. ASBESTOS DUST CONCENTRATIONS AND FIBER COUNTS

The principal sources of information on asbestos dust concentrations before the introduction of the membrane filter method are measurements by impinger in the United States going back to the 1930s,<sup>42</sup> and in Canada from 1944,<sup>43</sup> by konimeter and thermal precipitator in South Africa from 1940,<sup>44</sup> and by thermal precipitator in England from 1952.<sup>45,46</sup> Mostly, these early measurements were of particles without separate discrimination of fibers.

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It is important to remember that measurements of dust concentrations to which workers are exposed depend not only on the efficiency of the sampling instruments and sample evaluation, but also on the sampling strategy — when and where the samples are taken in a variable environment and among a moving work force. Procedures as well as instruments have changed over the years.

### 1. Impinger

The impinger was introduced in 1922 and the smaller more-convenient “midget impinger” was introduced in 1937.<sup>8</sup> It was customary to express particle concentrations in terms of millions of particles per cubic foot (mpcf). The impinger remained the standard method of dust measurement in the United States for 50 years, becoming entrenched because many hygiene standards and vast accumulations of data were based on it. Distinguishing features, compared to the konimeter and thermal precipitator, are that the dust is collected and examined in liquid (water or alcohol) leading to dispersal of aggregates; the sample can be diluted to optimum concentrations for evaluation; particles smaller than circa 0.75  $\mu$ m are not detected.

The counting of impinger samples is subject to observer differences (as found with other dust counts). Counts on the same sample by untrained personnel may differ by a factor of 10, and even experienced personnel can be expected to differ considerably, with variation by a factor of up to three between the lowest and highest counts of the same sample.<sup>47</sup>

Although the impinger was used to measure dust concentrations in the asbestos industry — and a TLV of 5 mpcf for asbestos was listed by the ACGIH from 1946 to 1970 — it does not provide a direct measure of asbestos fibers. The majority of fibers are not captured by the impinger because of their thinness and small aerodynamic diameter; even if captured, they are not seen in the counting process. Ayer et al.<sup>48</sup> found that in asbestos textile plants, where the only dust generated comes from asbestos, the total impinger particle count included less than 10% of fibers. South African workers<sup>49</sup> considered the impinger “completely unsuitable” for the measurement of asbestos because of the excessive failure to capture fibers 5 to 100  $\mu$ m long, as found by passing the effluent air through a membrane filter.

Nevertheless, some correlation between impinger dust counts and asbestos fiber concentrations during work with asbestos might be expected because both are reflections of the general dustiness of a work place and should rise or fall more or less together in response to changes in dust suppression measures, ventilation, etc. The relationship will, however, be dependent on the nature of the process and the composition of the source materials. The constancy of the size distribution of dust formed from a given material is not relevant to the relationship between fine fibers and coarser nonfibrous particles arising from disparate asbestiform and nonasbestiform materials. The relationship between impinger particle counts and PCOM fiber counts by membrane filter based on parallel measurements by the two methods has been investigated in the United States and Canada. An extensive study by the U.S. Public Health Service at a variety of asbestos processing plants<sup>48,50</sup> in the 1960s gave the ratios shown in Table 4. As might be expected, the fiber/particle ratio (FPR) is higher for asbestos textile manufacture, where the material processed is entirely “asbestos fiber”, than for friction materials or cement pipe, where the product has other constituents. At one of the chrysotile textile plants, there was also an extensive company sampling program in which further measurements were made with both impinger and membrane filter between 1965 and 1971; similar FPRs were obtained — the PCOM fiber concentrations corresponding to 1 mpcf by impinger were 8 fibers/ml (f/ml) for asbestos “preparation” and 3 f/ml for all “textile operations”.<sup>42</sup>

Gibbs and Lachance<sup>51</sup> reported paired measurements by impinger and membrane filter at seven Canadian chrysotile mines and mills, and Dagbert<sup>52</sup> obtained from them a nonlinear relationship between impinger PCOM fiber concentrations (f/ml) and particle concentrations (mpcf) with a mean FPR of 11.0 (f/ml)/(mpcf), but with extremely wide variation. In an epidemiological study at the above Canadian mines by Liddell et al.,<sup>53</sup> fiber exposures were

**TABLE 4**  
**Fiber Counts<sup>a</sup> Related to Concentrations<sup>b</sup> of Dust Measured**  
**by Midget Impinger in Asbestos Processing Plants, USA<sup>c</sup>**

Product	Sample pairs	FPR <sup>d</sup>
Textile	500	5.9
Friction	200	2.2
Asbestos cement pipe	100	1.9

<sup>a</sup> "PCOM fibers" per milliliter (f/ml).

<sup>b</sup> Million particles per cubic foot (mpcf).

<sup>c</sup> From Lynch et al.<sup>50</sup>

<sup>d</sup> Fiber particle ratio: (f/ml) for 1 (mpcf).

estimated from historic impinger counts by the use of conversion factors ranging from 0.3 to 40 (f/ml)/mpcf for different areas and subareas, but "averaging" around 3.5.

The 1968 British BOHS standard<sup>33</sup> for chrysotile asbestos dust (the first to be based on PCOM fibers) gave an "alternative index" for measurements by impinger that implied a FPR of 7. In 1968 to 1969, the ACGIH proposed alternative TLVs in terms of impinger particles and PCOM fibers (longer than  $>5 \mu\text{m}$ ) based on the relationship that 1 mpcf corresponds to 6 f/ml. The long-established impinger-based TLV was abandoned in 1974.

The impinger was not used extensively for dust measurement outside North America, and there appear to be no comparable data relating impinger mpcf to PCOM f/ml for varieties of asbestos other than chrysotile.

## 2. Konimeter

There are various forms of konimeters, but all have the same essential features. They are small hand-held instruments in which a 5 ml sample of air is drawn through a 0.5 mm diameter orifice in about 0.25 s by a spring-loaded pump and impinges against a greased glass slide on which dust particles are deposited by impaction. Dust collection efficiency varies with the nature of the dust, the thickness of the adhesive film on the slide, etc. The impaction process is vigorous and theoretically should be effective down to about  $0.5 \mu\text{m}$  for compact mineral particles and thereafter fall off rapidly; in practice, large particles (a few  $\mu\text{m}$  in size) may either bounce off the slide and escape capture or shatter and produce spurious small particles; also aggregates may be torn apart, giving an enhanced count. Overcrowding of particles in the center of the dust "spots" can make evaluation difficult (and possibly affect collection efficiency), so that sampling efficiency appears to vary with concentration. In South Africa, slides with dust deposits were usually heated and acid-washed before evaluation, to remove nonsiliceous dust particles.<sup>44</sup>

Dust determinations by konimeter were made in South African (crocidolite, amosite, and chrysotile) asbestos mines and surface plants from 1940 to 1965, after which the standard thermal precipitator (STP) became the principal means of measurement.<sup>44</sup> The STP had been used earlier for special studies, and several comparisons between it and the konimeter were made between 1940 and 1967.<sup>54</sup> Separate counts of particles and fibers were made. Before 1965 any particle with a length/width ratio of 2:1 or more was assumed to be a fiber; this was raised to 3:1 in 1965. No minimum length was specified. STP samples were counted in the same manner as konimeter samples until 1945, when light-field microscopy was substituted, followed in 1965 by counting down to a lower size limit of  $0.5 \mu\text{m}$ . From these tests, DuToit<sup>54</sup> concluded that "for practical application the instruments are comparable for [total] fibers down to the limit of detectability."

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**TABLE 5**  
**Proportions of Long Fibers (>5  $\mu$ m), Short Fibers, and Particles**  
**in South African Asbestos Dust Sampled by STP**

Asbestos variety	Long fibers (>5 $\mu$ m)	Short fibers	Particles
Chrysotile			
Surface	7	45	48
Underground	3	42	55
Amosite			
Surface	19	36	45
Underground	3	30	67
Cape crocidolite			
Surface	8	43	49
Underground	5	36	59
Transvaal crocidolite			
Surface	13	36	51
Underground	5	23	72

Adapted from DuToit, R. S. J. and Gilfillan, T.C., *Ann. Occup. Hyg.*, 20, 333, 1977.

**TABLE 6**  
**Membrane Filter Fiber Concentrations Compared with Count by Konimeter and**  
**STP, South African Asbestos Mines Surface Plants: Ratios of Mean Fiber Counts**  
**by Membrane Filter to Mean Fiber Counts by Konimeter and STP**

Asbestos variety	Konimeter		STP	
	1 (f/ml)	5 (f/ml)	1 (f/ml)	5 (f/ml)
Chrysotile	4.6	3.4	7.3	4.2
Amosite	4.4	4.2	5.7	9.8
Cape crocidolite	0.8	1.5	0.9	0.8
Transvaal crocidolite	2.2	2.6	1.9	1.8

Calculated from DuToit, R. S. J. and Gilfillan, T. C., *Ann. Occup. Hyg.*, 20, 333, 1977.

Table 5 gives the proportions of long fibers (>5  $\mu$ m), short fibers, and particles for different types of asbestos produced in South Africa, "determined by means of a thermal precipitator for a size down to about 0.5  $\mu$ m and for a large number of samples", as presented by DuToit at a symposium of invited international experts held in Johannesburg in 1977.<sup>55</sup>

The same symposium recommended that the membrane-filter technique should be adopted universally to allow comparison of the dust conditions in various countries and to relate present conditions with past conditions by means of appropriate conversion factors. In pursuance of this, tests were made with konimeter, STP, and membrane filter (8  $\mu$ m pore size), operated in parallel at surface (mill) and underground sites at eight mines representing four varieties of asbestos.<sup>56,57</sup> Only fibers, defined as particles of length 5 to 100  $\mu$ m, length/width ratio >3:1, and diameter <5  $\mu$ m, were counted. Despite reduction of the sampling rate when high dust concentrations were expected, there was very large variation in fiber density on the filters — from 126 to 18,000 f/mm. Although the results showed a wide spread, with milling operations producing much higher fiber concentrations than mining, statistically significant functional relationships between concentrations measured by the different methods were derived, from which the data in Table 6 are calculated.<sup>56</sup> Except for Cape crocidolite, the

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membrane filter method indicated higher concentrations than the older methods by amounts that varied between asbestos types and with concentration, up to almost tenfold.

Additional tests to compare fiber counts obtained with 8  $\mu\text{m}$  (Table 6) and 0.8- $\mu\text{m}$  pore size membrane filters were made in a laboratory dust duct for Cape crocidolite only.<sup>57</sup> Substantially higher counts were obtained with the finer filter to an extent that was out of line with experience elsewhere. If similar ratios were applicable to other asbestos varieties, as suggested by the authors, it would further increase the membrane filter counts relative to konimeter and STP. These somewhat bizarre results would seem to indicate variable underestimation of long fibers by konimeter and STP, due either to sampling inefficiency for such particles or to overlap errors in too-dense samples. (In an international exchange of membrane filter samples to compare counting levels carried out at about the same time, South African counts were near the overall mean.<sup>37</sup>)

A further study to link past measurement by konimeter and STP with present membrane filter results was carried out at South African Cape crocidolite (three) and amosite (one) mines and mills, in collaboration with German representatives.<sup>58</sup> The importance of fiber loading was recognized and sampling times were about 40 to 60 minutes as compared with 4 h in the previous study, giving densities mostly within recommended limits; STP times were 40 to 60 min as before; konimeter sample volumes were fixed by instrument design. The South African and German counts of the membrane filter samples were in satisfactory agreement, and — in contrast to the previous findings — results for the two varieties of asbestos were similar and could be combined; the membrane filter gave rather lower counts than konimeter or STP. With reference to the earlier findings,<sup>56,57</sup> the authors suggested<sup>58</sup> that the variations in relationships with concentration and asbestos variety might be associated with differences in sampling rates and consequent differences in filter loadings. However, the careful restriction of filter loading in the later work would have been expected to give higher fiber counts relative to konimeter and STP and not the observed reduction.

### 3. Thermal Precipitators

With the STPs, deposition is very efficient from about 5  $\mu\text{m}$  down to the smallest sizes; gravitational effects progressively interfere with the collection of larger (compact) particles, but it was reported that the instrument was not efficient in capturing "large fibers (above 10  $\mu\text{m}$ )".<sup>33</sup> Sampling time with STP must be adjusted to suit the dust conditions, as overlapping of deposited particles can lead to serious underestimation of particle concentrations if samples are too dense.

In the LRTP, the sampled air passed first through an elutriation chamber to remove nonrespirable particles and then over a sedimentation zone of the slide before reaching the thermal deposition zone. This spread the sample over a larger area and, with the lower flow rate, permitted sampling for much longer periods, compared to the STP, without overcrowding the sample.

For regular dust monitoring at an asbestos textiles factory at Rochdale, U.K., the STP was used during the early period, 1952 to 1960. The STP was replaced by the LRTP from 1961 until 1964, when the membrane filter method was introduced. The LRTP was used with the elutriator cover removed and was then found to be effective in sampling long fibers, unlike the STP. The samples were evaluated for fibers longer than 5  $\mu\text{m}$  by light-field microscopy. No comparisons between STP particle and LRTP fiber counts were made at the time of changing methods, but estimates of the FPR were made by the BOHS Committee on Hygiene Standards,<sup>33</sup> who compared process mean dust levels in 1960 (the last year of using the STP) and 1961 (the first year with the LRTP). The FPRs varied greatly, with an overall mean, weighted for the number of workers in each process, of 1 (fibers/ml): 38 (particles/ml). This is numerically equivalent to 1 (fiber/ml)/(ppcf), but it must be remembered that particle measurement by TP and impinger are not commensurable. The BOHS committee also considered that

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the LRTP and the membrane filter methods gave comparable results and that no conversion factor was required.

The Rochdale evidence was reexamined by the BOHS in 1983<sup>45</sup> in an exercise mounted to compare directly the various sampling methods employed at the factory, by using original equipment and calling on the assistance of personnel who had been involved in the original work. These results indicated that the ratios of (modern) membrane filter counts to the LRTP and STP counts had increased, as compared to the previous assessments, by factors of 2.2 and 2.5, respectively. It was noted that the trial was carried out in much lower dust concentrations than had prevailed in 1960 and that factory processes — and probably dust quality — had changed too.

#### 4. Conclusion

From the foregoing, it is clear that the problems of reassessing historic measurements in terms of modern techniques are almost insuperable. Indeed, Doll and Peto,<sup>46,59</sup> after full discussion of the problems, preferred to apply the 1960/1961 factors to the Rochdale data.

## VII. MEASUREMENT METHODS USED FOR MAN-MADE MINERAL FIBERS

Historically MMMF dusts have not been subject to extensive regulation but have been classed among the nuisance particulates to which the ACGIH assigned a TLV of 30 mpcf or 10 mg/m<sup>3</sup> total suspended particulates; most countries followed this, or used a respirable dust limit of 5 mg/m<sup>3</sup>.<sup>60</sup> Consequently, only limited records exist of past concentrations in terms of mpcf (in the United States) or total mass.

### A. MEMBRANE FILTER METHOD APPLIED TO MMMF

Following concern about a possible fiber hazard from MMMF analogous to that from asbestos, the membrane filter method has been used to measure fiber concentrations, both in the United States<sup>61,62</sup> and in Europe,<sup>63</sup> using protocols similar to those developed for asbestos. The dust measurements at 13 selected factories throughout Europe were made by the Institute of Occupational Medicine (IOM), Edinburgh, between 1977 and 1980, who also exchanged samples with national laboratories in the different countries to establish relationships between counting levels. After earlier experience with asbestos, it was no surprise to find up to threefold differences.<sup>64</sup> The development of a reference scheme analogous to that adopted for asbestos was therefore initiated by WHO (Europe), and after a series of laboratory workshops and slide exchanges the WHO/Euro Reference Scheme was published.<sup>3</sup> This is generally similar to that for asbestos but has rules for counting complex particles that lessen some of the difficulties found with those for asbestos. These developments substantially changed the counting levels of some participating laboratories. IOM counts were increased about three times, and the previously reported survey results were adjusted accordingly.<sup>65</sup> Overall, inter-laboratory variation was greatly reduced,<sup>64</sup> but the comparisons were not extended to the assessment methods used in the United States.

### B. USE OF GRAVIMETRIC AND IMPINGER MEASUREMENTS

Measurements of total or respirable mass concentration may provide a useful hygienic index for nuisance or fibrogenic dusts but are not directly relevant to the specific hazard from fibers. The airborne dust in MMMF manufacturing plants and in user situations contains a considerable proportion of nonfibrous material, some of which will be of the same composition as the fibers. The proportion of fibers (all sizes), not all of which is of dangerous respirable size, has been estimated as 0.5 to 20% by weight. Since mass measurements have been used